SCIENCE FOR CERAMIC PRODUCTION

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MOLDABILITY OF CERAMIC MOLDING POWDERS DEPENDING ON THE METHOD OF PREPARATION

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The properties of molding powders of aluminum-oxide material VK 94-1 (containing 95% Al_2O_3) produced by drying in a spray dryer and their effect on the properties of molded samples are investigated. The technological process of molding refractory saggers using clay preliminary dried in a spray dryer is analyzed. Recommendations for further improvement of properties of saggers are issued.

Products made of aluminum-oxide nonplastic material VK 94-1 (containing 95% Al_2O_3) are usually molded by compression. This is carried out using granulated molding powder produced by briquetting and subsequent crushing and screening on respective sieves.

The implementation of quasi-isostatic molding technology for a wide product range, such as vacuum-dense insulators shaped as smooth rings (diameters 86, 130, 180, 190, and 250 mm, height up to 170 mm), rings with a fin configuration of diameter 156 mm, and milling balls (diameters 20, 30, 40, 50, and 60 mm), altogether 13 products, initially relied on molding powders preparing by briquetting. Quasi-isostatic molding does not require special technology of powder preparation, and the same technology as in static molding is used. Due to the increasing demand for insulators (the weight of the molded preform for some types of insulators was 10.6 kg) the company changed to preparation of molding powders in a spray drier.

For this purpose a spray drier with an output not less than 250 kg/h was developed and brought into service [1].

It is known that stable molding processes and uniform density of the preform require good friability of molding powder, absence of refractioning, and constancy of its packing density inside the mold volume. These conditions are especially significant in the case of complex configurations of product and stringent requirements imposed on their properties, for instance, in the production of different types of technical ceramics [2].

However, molding articles from powder produced in a spray dryer involves difficulties caused by instability of powder properties. Therefore, it was necessary to perform additional studies of the moldability of such powders. The study investigated molding powders with a technological binder containing 4.0% polyethylene glycol and 0.7% polyvinyl alcohol, which was introduced into slip prepared for drying.

The properties of several batches of such powders and their moldability in metal molds were investigated (Table 1). The powders obtained had unstable properties. Thus, the moisture from one batch to another varied from 0.07 to 2.38%. The calcination losses varied over an inadmissibly wide interval, from 3.44 to 4.41%, which points to unstable drying conditions. Apparently, under certain spraying regimes a substantial quantity of the plasticizer burns out, which significantly deteriorates the moldability of powder. The highest bulk density (about 1.05 g/cm^3) was observed in the molding powder of moisture 2.38%.

The granulometric composition of molding powders also varied from one batch to another. Thus, the content of the fraction 500 – 250 μm varied from 13.68 to 24.24%, that of the fraction 250 – 100 μm from 72.24 to 61.36%, and that of the fine fraction (finer than 100 μm) from 16.68 to 9.32%. It is known that the presence of a certain quantity of fine particles in coarse powder increases its density as the fine particles fill the pores. However, the presence of a significant quantity of fine particles substantially complicates the removal of air from powder in molding. Furthermore, with an increasing specific surface area of powder, the friction against the mold walls and between the powder particles increases substantially [2]. Friction is one of the main factors affecting the process of powder molding. The force of friction impedes the transmission of pressure to the adjacent lay-

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TABLE 1

Sprayed powder batch*	Molding powder moisture, %	Calcination loss, %	Total content of technological binder, %	Bulk density, g/cm^3	Friability, g/sec	Granulometric composition, %, of particle size, μm					
						500	250	> 100	> 71	< 71	< 100
5/1	0.15	3.90	4.05	1.04	7.22	0.28	24.24	64.28	9.28	1.92	11.20
7/2	0.07	3.44	3.51	1.03	7.33	–	18.20	72.24	6.10	3.44	9.54
10/3	1.84	4.41	6.25	1.04	7.57	0.24	23.00	61.36	12.76	2.64	15.40
11/3	2.38	4.07	6.45	1.05	7.37	0.18	18.41	70.07	8.62	2.72	11.34
13/1	1.53	3.86	5.39	1.01	5.88	–	–	–	–	–	–
13/1 (granulated)	0.79	3.64	4.43	1.05	7.75	–	–	–	–	–	–
20/1	0.90	3.52	4.42	1.01	6.57	–	–	–	–	–	–
21/1	1.47	3.93	5.40	1.00	5.61	0.08	14.64	71.24	7.12	6.92	14.04
21/1 (after aging for 1 day)	0.52	3.90	4.42	1.02	7.14	0.24	19.18	66.56	6.08	8.00	14.08
21/2	0.76	3.70	4.46	1.04	7.17	0.12	13.68	69.52	11.96	4.72	16.68
23/2	0.37	4.14	4.51	1.04	7.46	0.16	19.44	71.08	4.92	4.40	9.32

* First digit) sprayed powder batch; second digit) number of the car of capacity 250 kg.

TABLE 2

Molding powder batch	Properties of molded samples			
	height, mm	compression coefficient	density, g/cm^3	moisture, %
From spray dryer:				
5/1	10.58	2.36	2.38	0.15
7/2	10.67	2.35	2.36	0.07
10/3	10.09	2.48	2.49	1.84
11/3	9.88	2.53	2.57	2.38
13/1	10.15	2.46	2.47	1.53
20	10.39	2.40	2.42	0.67
21/1	10.09	2.48	2.49	0.90
21/1 (after aging)	10.05	2.49	2.50	0.52
21/2	10.26	2.43	2.45	0.76
23/2	10.05	2.49	2.50	0.37
From briquetted	9.30	1.76	2.71	4.62
powders	9.32	1.76	2.70	4.53
	9.28	1.77	2.72	4.75

TABLE 3

Moisture of molding powder produced in spray dryer, %	Calcination loss, %	Shrinkage coefficient	Density of fired sample, g/cm^3
0.07	4.20	1.180	3.70
0.15	4.07	1.176	3.71
0.37	4.25	1.170	3.73
1.15	5.60	1.149	3.74
1.84	6.25	1.140	3.74
2.38	6.45	1.130	3.75

ers, which leads to a nonuniform pressure distribution across the product and, accordingly, to its nonuniform density. In this connection the content of the fine fraction in molding powder should be sharply decreased. Apparently the quantity of the 100-μm fraction should not exceed 2 – 4%.

The instability of molding powder properties was also registered within the same batch: for instance, in batch 21 the powder moisture on one car was 1.47% and on another car 0.76%. Furthermore, for certain powder batches, zones of different moisture, friability, and granulometric composition were identified within the same car, moreover; the powder on the bottom of the car had insufficient friability. Apparently its granules had insufficient mechanical strength and was destroyed under the pressure of the top layers.

The moldability of powders obtained by spraying was studied on samples of diameter 10 mm molded in metal molds at constant unit pressure equal to 100 MPa. The sample portion weight for all powders, including granulated ones, was taken equal to 1.98 g. Each powder was used to mold 10 samples.

The moldability was estimated based on the height and the volume weight of molded samples and the powder compression coefficient. A lower height of a sample indicates better moldability of the powder, as denser packing of particles is provided in the course of molding.

Table 2 gives an average height value for ten samples. Powder 11/3 had the best moldability of all powders produced in the spray dryer. The average height of the samples was 9.88 mm and approached the height of the samples molded from granulated powders. All molding powders produced by spraying had a high compression coefficient, which is evidence of loose packing of powder particles related to the presence of a great quantity of the fine fraction. After the fine fraction in powder 13 was screened, its bulk density increased.

The dependence of the fire shrinkage coefficient and volume weight of fired samples on molding powder moisture was established. The samples were molded at a unit molding pressure of 100 MPa (Table 3). It was established that with increasing moisture of molding powders produced by spraying their shrinkage coefficient decreases and with moisture 2.38% it is equal to the shrinkage coefficient of samples

made of granulated powder. The density of fired samples with increasing moisture grows from 3.70 to 3.75 g/cm^3.

The unit molding pressure required for samples from sprayed powder of moisture 0.2 – 0.6% was determined for the purpose of reaching a density equal to the density of the samples produced from granulated powders. The studies were performed on the molding powder of batch 23/2 with moisture 0.37%. For comparison purposes samples made from granulated powder were simultaneously molded at a unit pressure of 100 MPa.

TABLE 4

Molding powder batch	Unit molding pressure, MPa	Density of preform, g/cm^3	Height of molded samples, mm	Density of fired sample, g/cm^3	Height shrinkage coefficient
23/2	100	2.50	10.33	3.69	1.16
23/2	150	2.57	10.01	3.71	1.14
23/2	200	2.68	9.68	3.73	1.13
Samples from briquetted powders	100	2.71	9.42	3.74	1.13

It was found that the sprayed powder of moisture 0.37% requires a molding pressure of 200 MPa, i.e., two times higher (Table 4). However, an increased molding pressure requires a fortified mold design and shortens the mold service period. Therefore, it is advisable to perform molding with a higher moisture of powder, but at a lower pressure level. The data in Table 3 indicate that the same density and the fire shrinkage coefficient can be reached under a unit pressure of 100 MPa using sprayed powder of moisture 2.38%.

Consequently, the moisture of powders produced by spraying should be no less than 2.5%.

The study of the moldability of the aluminum-oxide material indicated that molding powders produced by drying in a spray dryer do not fully meet the moldability requirements. The powders have unstable moisture, calcination losses, and granulometric composition and require an increased molding pressure. The density of a molded preform increases with increasing moisture.

A hard surface of granules intensifies the wear of molds. An increased molding pressure calls for a fortified mold design, increased wear resistance, and more powerful machinery.

It should be noted that clay preliminary dried in a spray dryer has a similar effect on the quality of refractory saggers.

Analysis of the serial process of molding refractory saggers at ceramic works indicated that one of the reasons for their inadequate quality is heterogeneity of sagger mixtures caused by using clay preliminarily dried in a spray dryer [3]. The high temperature inside the drying chamber results in the formation of hard granules. Consequently, the time allowed for mixing chamotte mixtures in the technological process is obviously insufficient. Therefore, the prepared mixtures have decreased plasticity, and the molding pressure has to be increased to obtain a required preform density [4].

Thus, the spray dryers operating at mass-production works do not fully provide for the production of molding powders with required molding properties. In this context, in the case of preparing molding powders by drying in a spray dryer, the dryer design should be selected and adjusted with a view to lowering the temperature inside the drying chamber, increasing the content of the technological binder in the powder, and producing molding powder granules whose composition is homogeneous to the composition of the technological binder.

REFERENCES

1. M. I. Timokhova, “Quasi-isostatic molding in serial production of ceramics,” *Steklo Keram.*, No. 8, 14 – 19 (2002).
2. R. Ya. Popil’skii and V. F. Kondrashov, *Molding Ceramic Powders* [in Russian], Metallurgiya, Moscow (1968).
3. M. I. Timokhova and V. S. Rachkov, “Quasi-isostatic molding of refractory saggers,” *Steklo Keram.*, No. 3, 22 – 24 (2002).
4. M. I. Timokhova and L. I. Medvedeva, “A highly efficient process of molding miniature ceramics,” *Steklo Keram.*, No. 8, 13 – 15 (2003).